

Spin Dynamics at the Mott Transition and in the Metallic State of the Cs_3C_{60} Superconducting Phases

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Abstract. - We present here ^{13}C and ^{133}Cs NMR spin lattice relaxation T_1 data in the A15 and fcc- Cs_3C_{60} phases for increasing hydrostatic pressure through the transition at p_c from a Mott insulator to a superconductor. We evidence that for $p \gg p_c$ the $(T_1T)^{-1}$ data above T_c display metallic like Korringa constant values which match quantitatively previous data taken on other A_3C_{60} compounds. However below the pressure for which T_c goes through a maximum, $(T_1T)^{-1}$ is markedly increased with respect to the Korringa values expected in a simple BCS scenario. This points out the importance of electronic correlations near the Mott transition. For $p \gtrsim p_c$ singular T dependences of $(T_1T)^{-1}$ are detected for $T \gg T_c$. It will be shown that they can be ascribed to a large variation with temperature of the Mott transition pressure p_c towards a liquid-gas like critical point, as found at high T for usual Mott transitions.

Introduction. – Among the various systems presenting a transition from a magnetic to a high T_c superconducting (SC) state, the recently discovered fulleride A15- Cs_3C_{60} phase [1] takes a very special place. Indeed, as in cuprates, the transition observed there is from a Mott insulating state to a SC state, contrary to Fe pnictides in which SC and metallic magnetism are contiguous. We have found [2] that the pressure controlled transition occurs at different pressures p_c of 3.5(5) kbar and 6.5 (5) for the two isomeric structures of Cs_3C_{60} (A15 and fcc respectively). Below p_c the ground magnetic states of the insulating phases were found distinct for the two structures. The A15 is a Néel antiferromagnet (AF) while the fcc phase, which is not a bipartite structure, displays a more complicated magnetism. Otherwise the phase diagrams can be mapped into a unique one, recalled in Fig. 1, when plotted versus $V_{\text{C}_{60}}$, the unit volume per C_{60} molecule (as confirmed independently in Ref. [3]). This established that the interball distance is the driving parameter for the electronic properties of both compounds. The phase diagram displays a SC dome, reminiscent of that found in cuprates, with a T_c maximum near the first order phase boundary below which the insulating Mott state sets in.

But this analogy should be taken cautiously, as in the fullerenes the transition occurs in a three-dimensional (3D) crystal structure with a fixed number of carriers, while in cuprates it appears in a two-dimensional (2D) lattice of Cu sites, with hole or electron doping [4], which is furthermore a source of dopant induced disorder of prime importance for the physical properties [5]. More importantly A_3C_{60} have been so far considered as standard BCS(-type) phonon driven superconductors [6], with a clear singlet s -wave pairing, distinct from the d -wave pairing demonstrated in cuprates [7] or anticipated in 2D organics [8]

One should notice that such a phase diagram had been anticipated by Capone *et al.*, from ground state calculations assuming an electron phonon mechanism for SC [9–11]. The electronic correlations are of course embedded in this theoretical treatment in which a low spin $S = 1/2$ state is favoured by the on-ball Jahn-Teller effect for the three electrons transferred on the C_{60} balls. These authors predicted that pure BCS equations cannot apply and that the normal state near the Mott transition, where T_c increases with pressure, is a non Fermi liquid exhibiting a moderate increase of the spin susceptibility or effective mass.

Here we report NMR data on the electronic properties of these compounds which can be compared to these pre-

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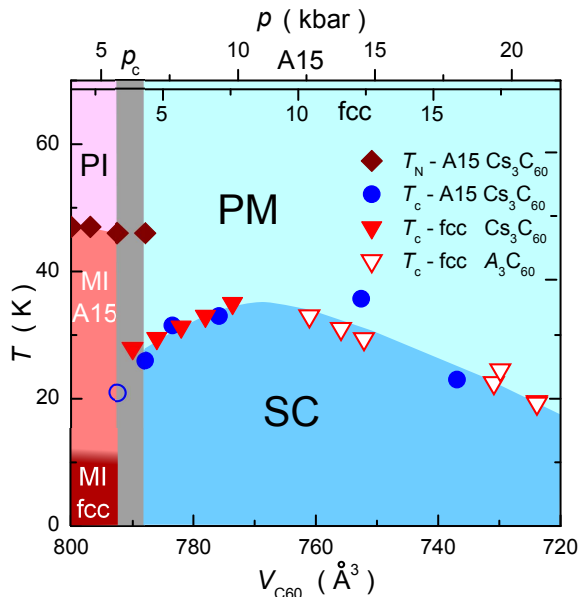


Fig. 1: (colour on line) Phase diagram representing T_c versus the volume $V_{C_{60}}$ per C_{60} ball, and the Mott transition at p_c (hatched gray bar). The magnetic insulating (MI) phase is AF with $T_N = 47$ K for the A15 phase. For the fcc phase a less characterized magnetic state occurs below 10 K. The corresponding pressure scales for the two phases are shown on the upper scale.

dictions. We demonstrate that the nuclear spin lattice relaxation T_1 data on ^{13}C and ^{133}Cs taken near the Mott transition can by no way be described by a simple pressure induced modification of the density of states in a non correlated band. On the contrary magnetic fluctuations concentrate near the Mott transition and only progressively give place to those resembling a Fermi liquid through the pressure for which T_c is maximum. We give here evidence for a step increase of T_1^{-1} at the critical pressure p_c , a behaviour quite analogous to that seen in organic compounds displaying a Mott transition [12]. We find that p_c increases with T as expected from the lattice expansion. Furthermore $p_c(T)$ apparently terminates at a critical point located near room temperature.

Experimental techniques. — One only expects limited accuracy from ^{13}C NMR shift measurements in fullerene compounds in view of the large anisotropy of hyperfine couplings for the ^{13}C nucleus [13]. We could however evidence that way [2] the large paramagnetic response in the Mott state of the Cs_3C_{60} phases. But, in fullerene compounds, T_1 data have been generally more useful [14, 15].

Let us recall that T_1 allows to probe the wave vector \mathbf{q} dependent dynamic spin susceptibilities $\chi(\mathbf{q}, \omega)$ of the electron spins through the general relation

$$\frac{1}{T_1 T} = \frac{2k_B}{(\gamma_e \hbar)^2} \sum_{\mathbf{q}} [A_{\text{hf}}(\mathbf{q})]^2 \text{Im} \left[\frac{\chi(\mathbf{q}, \omega)}{\omega} \right], \quad (1)$$

Here the \mathbf{q} dependence of the hyperfine coupling constant $A_{\text{hf}}(\mathbf{q})$ of the probe nucleus is determined by its spatial location with respect to the magnetic sites.

We have therefore undertaken some ^{13}C NMR T_1 measurements on both A15 and fcc phases, as this allows comparison with the available data for the other alkali $A_3\text{C}_{60}$ fullerenes [13, 16], the hyperfine coupling being then local, i.e. independent of the lattice structure. We have selected here two samples described in Ref. [2] with respective C_{60} molecular concentration of A15, fcc and Cs_4C_{60} phases of (A1: 41.7 %, 12 %, 46.5 %) and (F1: 34 %, 55 %, 11 %). This allows us avoiding contamination by Cs_4C_{60} for the dominant fcc phase sample and by the fcc phase for the dominant A15 phase. We have also taken advantage of the selectivity permitted by the ^{133}Cs NMR [2], with three resolved Cs sites (O, T, T'), to study the T_1 variations in a large (p, T) range on sample F1. Furthermore, in this case the absence of quadrupole effects yields exponential recoveries of the nuclear magnetization.

The pressure experiments on the fcc phase were not performed using a classical clamp cell which might yield inaccurate pressure control as we are lacking temperature insensitive pressure sensors. Here we used an externally controlled system in which a pressure generator is connected to the pressure cell through a capillary tubing. The pressure is transmitted to the sample space via a pressure multiplier mounted in the pressure cell. We have used isopentane and Flourinert-77 (3M) as pressure media for the first and second stage respectively. Within this media setup we were able to accurately adjust the pressure which is measured at room T while cooling the cell, until the complete solidification of Flourinert. The pressure loss due to the multiplier has been calibrated and does not exceed 0.3 kbar in the pressure range used in this study, so that the accuracy of the pressure in these experiments is better than ± 0.15 kbar.

Spin dynamics in the metallic state. — In weakly correlated elemental metals, $\text{Im}\chi$ is related to $\rho(E_F)$, the density of states at the Fermi level, and A_{hf} is \mathbf{q} independent for a purely local on-site hyperfine coupling. Equation (1) resumes in that case into the Korringa relation

$$\frac{1}{T_1 T} = \frac{\pi k_B}{\hbar} [A_{\text{hf}} \rho(E_F)]^2. \quad (2)$$

Such a variation is of course not observed in the Mott insulating paramagnetic state. Indeed there, as can be seen for instance in Fig. 2 for the A15 phase, $(T_1 T)^{-1}$ increases markedly at low T for both ^{13}C and ^{133}Cs nuclei and drops abruptly at the Néel transition at $T_N = 47$ K. At the highest pressures, well into the metallic phase, the $(T_1 T)^{-1}$ data display the constant expected metallic behaviour. But, when p is decreased towards $p_c \approx 6.5$ kbar, the metallic behaviour is seen only above T_c up to about 100 K and deviations begin to occur at higher T , as will be much better seen on the fcc phase data.

Let us consider first here the ^{13}C NMR data, for which

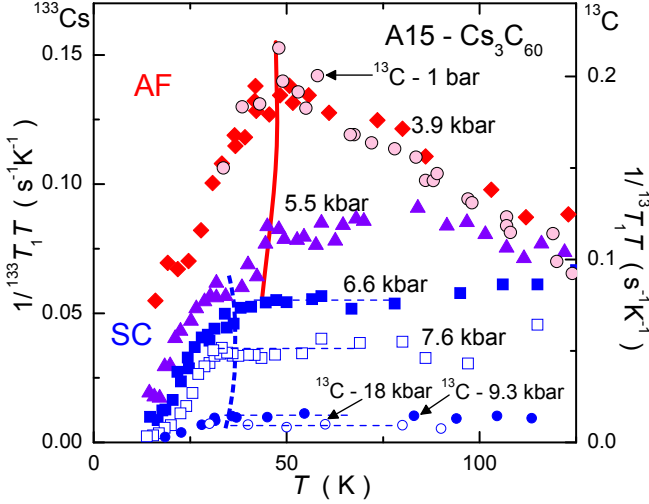


Fig. 2: (colour on line) In the A15 phase, $(T_1T)^{-1}$ has a large T variation in the paramagnetic insulating state at $p = 1$ bar and drops at the AF transition (full thick red line). At high p a constant Korringa like behaviour is seen above T_c , with the expected drop below T_c (thick dotted blue line) due to the opening of the SC gap. Notice the large increase of the "Korringa" constant (dotted thin line) when approaching the ≈ 6.5 kbar Mott transition. The ^{13}C data (circles) and the ^{133}Cs data have been scaled as discussed in the text.

these constant $(^{133}\text{T}_1T)^{-1}$ values taken well above the Mott transition pressure p_c are plotted then versus $V_{\text{C}_{60}}$ in Fig. 3. There, one can see that for both the A15 and fcc- Cs_3C_{60} phases the $^{13}\text{T}_1$ data merge with the smooth curve found on the other fcc- A_3C_{60} compounds [16].

The corresponding variation of T_c versus $V_{\text{C}_{60}}$ (or lattice constant) has been used at length in the past to indicate that a BCS formalism applies to the fcc- A_3C_{60} compounds. In this framework, T_c is given by

$$k_B T_c = 1.14 \hbar \omega_D \exp(-1/V\rho(E_F)). \quad (3)$$

This has led to consider that the Debye frequency ω_D and the electron-phonon coupling V depend solely on C_{60} molecular properties, so that a smooth variation of $\rho(E_F)$ with $V_{\text{C}_{60}}$ drives both variations of T_c and $(^{133}\text{T}_1T)^{-1}$ [6]. Here we observed by r.f. susceptibility measurements that T_c goes through a maximum versus $V_{\text{C}_{60}}$ in both phases while $(^{133}\text{T}_1T)^{-1}$ steadily increases. This is indicative of a breakdown of Eq. (3), as one would expect a decrease of $\rho(E_F)$ or of $(^{133}\text{T}_1T)^{-1}$ when T_c goes through the optimum value.

To better probe the evolution near the MIT, we considered as well ^{133}Cs data, which appear qualitatively similar to those on ^{13}C , as shown in Figs. 2 and 4. The raw data are found to display a large increase of $(^{133}\text{T}_1T)^{-1}$ with decreasing pressure from the top of the SC dome towards the Mott transition. This definitely points out an incidence of electronic correlations.¹ The ^{133}Cs data for

¹When magnetic correlations between C_{60} sites enter into play

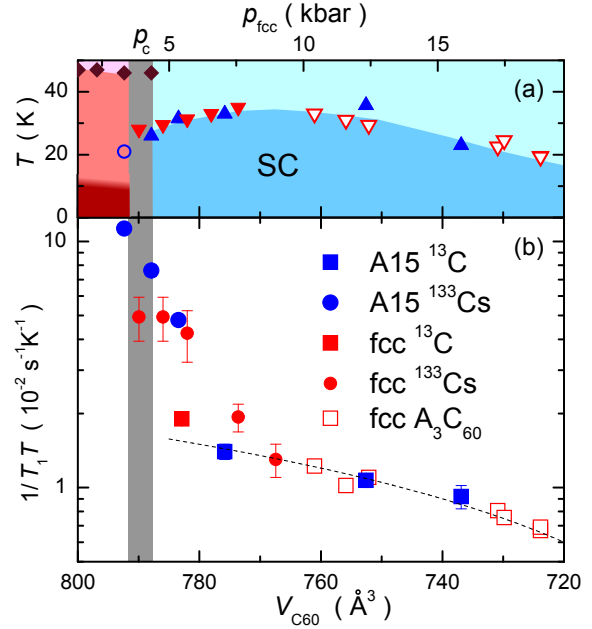


Fig. 3: (color on line) (a) The phase diagram of Fig. 1 is reproduced here as a reference, with the upper pressure scale for the fcc phase. (b) Plot versus $V_{\text{C}_{60}}$ of the ^{13}C "Korringa" $(^{13}\text{T}_1T)^{-1}$ value taken just above T_c in the metallic phases. Here the ^{133}Cs data displayed have been rescaled for comparison (see text). For both phases the simple BCS analysis of T_c , shown by a dotted line breaks down near p_c .

the A15 phase could be scaled with that of ^{13}C , using the ratio 1.4(2) obtained from the $p = 1$ bar data in the paramagnetic state from Fig. 2. For the fcc phase the ^{133}Cs T' site data plotted in Fig. 3 have been scaled by fixing the 9 kbar point of Fig. 4 on the dotted curve known for the smaller $V_{\text{C}_{60}}$ values. The scaled $^{133}(\text{T}_1T)^{-1}$ values in Fig. 3 display a large deviation near the Mott transition which confirms that Eqs. (2) and (3) do not apply.

Metal Insulator transition. — Let us now consider our measurements versus T for various pressures which we have performed mostly on ^{133}Cs NMR in order to benefit from the phase selectivity allowed by the large difference of spectra between the two phases on these nuclei (see Ref. [2]). The data displayed in Fig. 2 and Fig. 4 are qualitatively similar for both phases. However in the following part of the paper we shall restrict to the fcc phase on which we could take reliable data in a large range of pressures and temperatures. There we have measured T_1 on the three Cs sites O, T, T' but did find that at low T the signals of the O and T sites become so close in fre-

near the MI transition or near the Mott transition, the non local ^{133}Cs hyperfine coupling $A(\mathbf{q})$ might yield differences between the T variations of ^{133}Cs data and of the ^{13}C T_1 data, as the latter probe directly the on-site magnetic fluctuations. However, this may only yield a reduction of the contribution of the AF fluctuations to $(^{133}\text{T}_1T)^{-1}$, so the actual increase of $\text{Im}[\chi(\mathbf{q}, \omega)]$ when approaching p_c could only be larger.

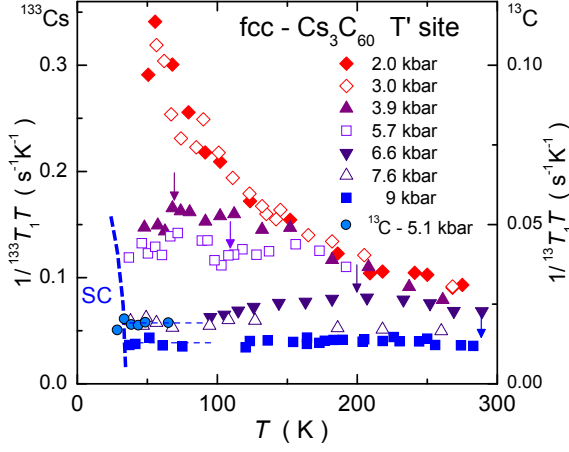


Fig. 4: (color on line) $(^{133}\text{T}_1T)^{-1}$ for the T' site of the fcc-phase. For each pressure above $p_c = 3.5$ kbar, the data have been taken above T_c which is delineated by the thick dotted blue line. $(T_1T)^{-1}$ departs with increasing T from the constant Korringa value (thin dotted blue line), goes through a maximum and decreases then towards the data obtained in the paramagnetic Mott state. The arrows are the positions of $p_c(T)$ determined as explained later in Fig. 5 and 6.

quency that they can hardly be resolved. This becomes even impossible below T_c due to the loss of spin susceptibility associated with singlet pairing. So we restricted the data displayed in Fig. 4 to that above T_c for the T' site which is better separated from the T site and has a T_1 close to that of the T site, which ensures that nuclear-spin cross relaxation effects do not influence significantly the data.

For $p > p_c$, the $(^{133}\text{T}_1T)^{-1}$ data differ markedly at low T from that in the Mott state, go through a maximum, and then decrease progressively at high temperature, as can be seen in Fig. 2. The regime of constant $(^{133}\text{T}_1T)^{-1}$, which extends nearly to room T in the higher pressure cases, reduces to smaller T ranges when p decreases towards p_c . This maximum in $(T_1T)^{-1}$ might bear some analogy to that seen in underdoped cuprates, and could be hastily attributed to a pseudogap [17], or spin gap in the magnetic excitations [18]. This maximum occurs at a temperature T^* which increases when p (and T_c) increase on the superconducting dome. Then, this analogy does not hold as T^* in cuprates decreases with increasing doping [17] towards the optimum T_c .

More importantly, one can notice in Fig. 4 that the $(^{133}\text{T}_1T)^{-1}$ data for $p > p_c$, after reaching their maximal values, evolve progressively at high T towards the $(^{133}\text{T}_1T)^{-1}$ data measured below p_c in the Mott insulating paramagnetic state. There the localized electron spins exhibit Curie-Weiss behaviour and for a dense local-moment paramagnet, one expects local fluctuation of the moments dominated by the exchange coupling J with their z neigh-

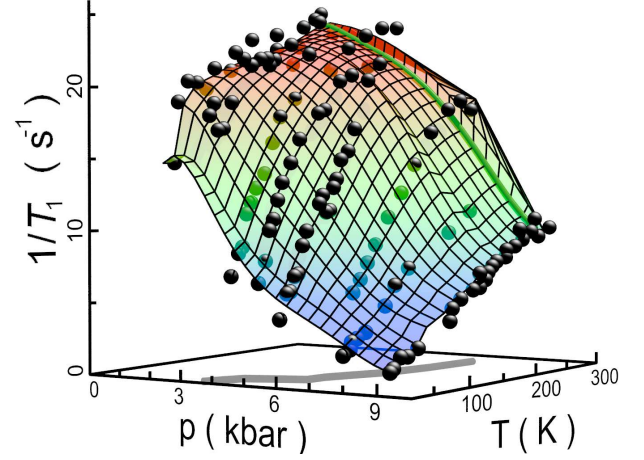


Fig. 5: (color on line) The T_1^{-1} data for the ^{133}Cs T' site of fcc- Cs_3C_{60} reported in Fig. 4 are shown here in a 3D plot versus (p, T) . The data can be approximated by the smoothed surface represented as a coloured grid, the black dots being above the grid while those below appear coloured, being seen by transparency. The transition $p_c(T)$ from the low p insulating state constant T_1^{-1} above 100 K to the high p nearly T linear behaviour in the metallic state is evident. The transition occurs at $p_c(T)$, projected in the ground plane, which shifts towards higher p_c with increasing T . A full line at $T = 250$ K gives an example permitting to view the inflexion point in the variation of $1/T_1(p)$ at $p_c = 6.6$ kbar, which corresponds to the arrow shown in Fig. 4 for the corresponding isopressure data.

bors, with a high temperature limit

$$\frac{1}{T_1} = [A_{\text{hf}}]^2 \frac{\sqrt{\pi S(S+1)}}{\sqrt{3}\gamma_e \mu_B k_B z J}. \quad (4)$$

This fits nicely the previous observations in A15 and fcc phases [2] for which $(T_1T)^{-1}$ scales at high T with T^{-1} [2, 19]. Then, in the fcc phase for pressures between $p_c = 3.5(5)$ kbar and 6.6 kbar, we see in Fig. 4 that at high T the $(T_1T)^{-1}$ data seems to keep initially the same behaviour as that found in the Mott state. At lower T the excitations are progressively suppressed and transform into the Korringa like behaviour discussed above in the metallic state. ²

To monitor differently the evolution of the local moment contributions to T_1^{-1} with increasing pressure we have therefore plotted the data of Fig. 4 in a 3D plot of T_1^{-1} versus (p, T) shown in Fig. 5. One can see as well that at high T the data converges for all pressures below 7 kbar towards the constant value found in the ambient pressure insulating state. Here one can see that the data below $p_c = 3.5$ kbar are nearly constant at high T . The decrease detected below 100 K has been assigned [2] to

² This points again at a different situation from that found for the cuprate pseudogap. Indeed in that case the pseudogap is a depression at low T of the DOS in a high T metallic state, while here the depression occurs in a Curie paramagnetic state and results in a low T metallic state.

the incidence of interactions between the local moments. This leads to a decrease in $(^{133}\text{T}_1)^{-1}$ much weaker in the frustrated magnet fcc phase [2] than in the real gap opening Néel phase of the A15- Cs_3C_{60} . In this representation one is then led to view isothermal lines, in which the T_1^{-1} data, constant at low p , in the insulating state drops with increasing p towards the metallic behaviour. Indeed if we look at the surface which approximates all the data points in Fig. 5, the drop at p_c from the low p to the high p behaviour at constant T and from the high T to the low T behaviour at constant p can be seen to occur at inflexion points T_{MIT} of the fitting grid. Those correspond in the latter case to the maxima indicated by arrows in Fig. 4. We do not see here very sharp steps of T_1^{-1} as would be expected for a first order transition. This appears quite natural as no special care was taken to see hysteresis effects in these isopressure measurements when varying the temperature. The large set of data obtained here however allows us to locate the transition pressure $p_c(T_{\text{MIT}})$ at which such a step like decrease of T_1^{-1} is observed at fixed $T = T_{\text{MIT}}$.

This reveals that the transition pressure p_c indeed increases with increasing T so that the $T_{\text{MIT}}(p_c)$ line is not vertical in the (p, T) plane, as shown in Fig. 6(a). One should however remind that these materials are quite compressible and have a high thermal expansion, the dilatation from 0 to 250 K corresponding to a 15 % increase of $V_{\text{C}_{60}}$. Therefore the data plotted in Fig. 5 correspond to variable inter-ball distance. If one takes into account the variation of the lattice constant with p and T from Ref. [3], one can as well plot the Mott transition temperature T_{MIT} versus $V_{\text{C}_{60}}$ as done on Fig. 6(b). It is remarkable then to find out that this leads to a nearly vertical line, which indicates that the transition is indeed governed by interball distance, that is by the magnitude of the transfer integral or U/W ratio.

Let us point out that a decrease of step height with increasing T could be guessed in the set of data of Fig. 4 and 5. However these data being only taken at that time for a limited set of pressure values we could not quantify the step height values accurately. To get better data on the evolution of T_1^{-1} at the transition we have then performed a more systematic study of its variation with p first at a fixed $T = 290$ K and then at $T = 100$ K. Here we do find now as seen in Fig. 6(c) that at 100 K the step of T_1^{-1} is quite visible and occurs at 4 kbar near the 3.5 kbar value obtained from the diamagnetic susceptibility data [2]. At 290 K the variation of T_1^{-1} is rather smooth so that the transition is more difficult to locate. The lower p arrow in Fig. 6(c), would correspond to a widened transition, while the second one would correspond to a crossover resuming in a kink at 7 kbar. Both cases would indicate that one approaches a critical point at room T , as in a liquid-gas transition ³, in analogy to what has been seen in other

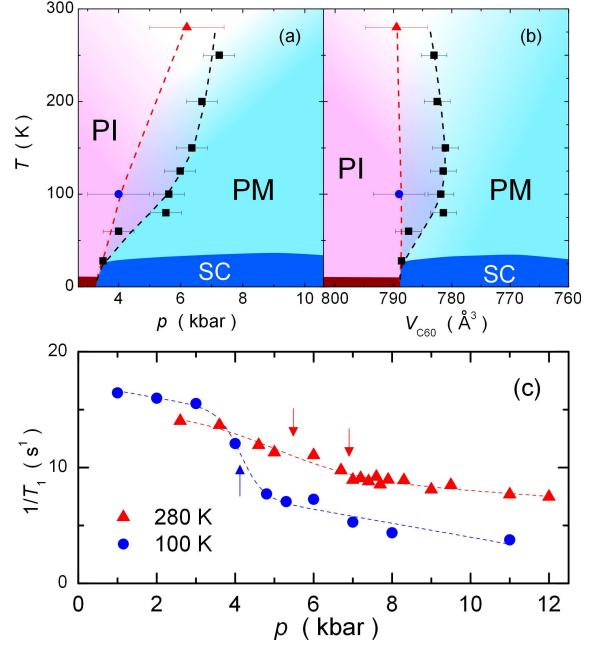


Fig. 6: (color on line) (a) Variation of T_{MIT} versus p_c obtained from the analysis of the two sets of experiments of Fig. 5 (squares) and of (c) below (circles and triangles). The various phases of the low T phase diagram of Fig. 1 are reproduced. (b) T_{MIT} is found nearly independent of $V_{\text{C}_{60}}$ after correcting for lattice volume variations. (c) Systematic $T_1^{-1}(p)$ data taken on 100 K and 290 K isotherms show that the T_1^{-1} step at $p_c(T)$ decreases markedly at room T (for the arrows: see text) which would be near the critical point for this fcc phase sample (all dotted lines in the three panels are guides to the eye).

Mott transitions i.e. in organic compounds [12].

Finally, as had been suggested for the metallic state of the dense $A_3\text{C}_{60}$ compounds [15], excitations from the $S = 1/2$ to the $S = 3/2$ Jahn Teller molecular state are expected at sufficiently high T . Those should be seen already in the insulating state and should appear as a high T increase of T_1^{-1} , as expected from Eq. (4) for $S = 3/2$. As seen in Fig. 5 we did not detect any such increase at $p = 1$ bar for fcc- Cs_3C_{60} , so that these excitations would only be detectable above room T . Capone *et al.* predicted that the energy distance between the two molecular states should decrease in the metallic state below the optimal T_c , yielding a situation reminiscent to that of a pseudogap. So far, the variation with pressure of the transition line does not allow us here to probe these excitations, which apparently might only occur above room T , even in the metallic states.

Discussion. — Here we have performed ^{133}Cs and ^{13}C T_1 measurements under pressure which bring quite novel

a slight decrease of T_1^{-1} values as compared to those of Fig. 5 has been observed together with a small shift of the transition towards lower pressures displayed in Fig. 6a. Such an evolution might be assigned to a small increase of sample metallicity, that is an increase of sample density at ambient pressure.

³The data of Fig. 6(c) has been taken six month later than Fig. 4 and 5. After curing such a long time at room T in the pressure cell

insight on SC near the Mott transition at p_c in the fcc- A_3C_{60} compounds, and on the Mott transition itself in the fcc phase of Cs_3C_{60} . We have shown that, just above T_c in the metallic state, $(T_1T)^{-1}$ deviates in both phases near p_c from the trend observed for the dense A_3C_{60} compounds. The large observed increase of $(T_1T)^{-1}$ indicates that spin fluctuations in the metallic state become prominent before the system switches into the Mott state.

Capone *et al.* [10, 11] were suggesting a moderate increase of the spin susceptibility when approaching the MIT, which would lead to deviations from the smooth T_c versus DOS BCS prediction. The significant increase of $(T_1T)^{-1}$ found here is similarly an evidence that the T_c variation deviates then from pure BCS but it is also a signature of strong magnetic correlations near the transition. Calculations of the dynamic susceptibility within their theoretical scheme should help to decide then whether the large T_c values in A_3C_{60} compounds does indeed result from a fundamental cooperation between spin fluctuations and electron phonon interactions.

We have shown here that the variations of $(T_1T)^{-1}$ versus T at fixed p although analogous to those expected for a pseudogap, mainly monitor the shift of the transition pressure with temperature due to lattice expansion. The large T dependence of the transition pressure p_c found here is somewhat similar to that seen in 2D-organics. In the present experiments, the "liquid-gas like" critical point of the phase diagram is located near room T . While in the 2D organics the Mott transition might be influenced by the pressure induced increase of interplane coupling, in the present 3D system we are probing a simple variation of U/W through the reduction of lattice parameter. Therefore, in the (p, T) range probed here the qualitative behaviour detected appears quite similar to those expected for a single orbital Mott transition [20], although the $S = 1/2$ ground state is orbitally degenerate.

To conclude, the local probe studies of Cs_3C_{60} provided here reveal that it is certainly a unique system in which 3D multiorbital behaviour coexists with singlet s wave fully gapped SC and spin fluctuations in the metallic state near the MIT. The fact that we could not evidence here the excitations between the low spin and high spin molecular states calls for ab initio determination of their energy difference. Measurements of other thermodynamic and spectral properties, possibly on larger T ranges are required to complete the experimental insight on this Mott transition.

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